



---

*Institute of Paper Science and Technology  
Atlanta, Georgia*

---

**IPST Technical Paper Series Number 949**

Sensor for Microstickies

J. Koskinen, D. Sung, F. Kazi, J. Yang, and S. Banerjee

July 2002

Submitted to  
TAPPI JOURNAL

*Copyright® 2002 by the Institute of Paper Science and Technology*

*For Members Only*

## INSTITUTE OF PAPER SCIENCE AND TECHNOLOGY PURPOSE AND MISSIONS

The Institute of Paper Science and Technology is an independent graduate school, research organization, and information center for science and technology mainly concerned with manufacture and uses of pulp, paper, paperboard, and other forest products and byproducts. Established in 1929 as The Institute of Paper Chemistry, the Institute provides research and information services to the wood, fiber, and allied industries in a unique partnership between education and business. The Institute is supported by 40 member companies. The purpose of the Institute is fulfilled through four missions, which are:

- to provide a multidisciplinary graduate education to students who advance the science and technology of the industry and who rise into leadership positions within the industry;
- to conduct and foster research that creates knowledge to satisfy the technological needs of the industry;
- to provide the information, expertise, and interactive learning that enable customers to improve job knowledge and business performance;
- to aggressively seek out technological opportunities and facilitate the transfer and implementation of those technologies in collaboration with industry partners.

## ACCREDITATION

The Institute of Paper Science and Technology is accredited by the Commission on Colleges of the Southern Association of Colleges and Schools (1866 Southern Lane, Decatur, Georgia 30033-4097; Telephone number 404-679-4501) to award the master of science and doctor of philosophy degrees.

## NOTICE AND DISCLAIMER

The Institute of Paper Science and Technology (IPST) has provided a high standard of professional service and has put forth its best efforts within the time and funds available for this project. The information and conclusions are advisory and are intended only for internal use by any company who may receive this report. Each company must decide for itself the best approach to solving any problems it may have and how, or whether, this reported information should be considered in its approach.

IPST does not recommend particular products, procedures, materials, or service. These are included only in the interest of completeness within a laboratory context and budgetary constraint. Actual products, materials, and services used may differ and are peculiar to the operations of each company.

In no event shall IPST or its employees and agents have any obligation or liability for damages including, but not limited to, consequential damages arising out of or in connection with any company's use of or inability to use the reported information. IPST provides no warranty or guaranty of results.

The Institute of Paper Science and Technology assures equal opportunity to all qualified persons without regard to race, color, religion, sex, national origin, age, disability, marital status, or Vietnam era veterans status in the admission to, participation in, treatment of, or employment in the programs and activities which the Institute operates.

## SENSOR FOR MICROSTICKIES

Jere T. Koskinen, Dong-Jin Sung, Feroz Kazi, Ji Yang, Sujit Banerjee

Institute of Paper Science and Technology  
500 10th Street, NW  
Atlanta, GA 30318

### SUMMARY

Stickies can cause major operational problems, and their detection and control has long been an industry priority. While several methods for determining macrostickies – adhesives caught in a 0.006" screen – are available, a rapid, consistent procedure for microsticking analysis is unavailable. It is proposed that the high molecular weight (>3,000 Da) colloidal material in process streams of secondary fiber mills is mainly microstickies. This material can be determined by first filtering a sample and measuring its total organic carbon (TOC) content. The filtrate is then ultrafiltered through a membrane that only allows passage of compounds of MW < 3,000 Da. The TOC of this low-MW permeate is then obtained. The difference in TOC represents high-MW (>3,000 Da) material, which corresponds to microstickies. A sensor that makes the entire measurement in eight minutes has been built for online use. The technique has been validated by comparison with two independent techniques using lab-derived samples as well as those collected from several points at six mills.

**Application statement:** An online microstickies sensor can be used to alert the mill of a high incoming microstickies load, test strategies to remove microstickies, and serve as a quality control tool.

---

### INTRODUCTION

Stickies can cause major operational problems, and their detection and control has long been an industry priority (1). Stickies are classified as microstickies if they pass through a 0.006" screen; larger particles are termed macrostickies. Several methods for determining macrostickies are available (2). Typically, the contaminants are isolated through screening, transferred to a sheet and imaged (2-9). Methods reported for quantifying microstickies include the Berol/polyethylene film method (10), the polyethylene bottle technique (11), the Doshi procedure (12), the Pira papermachine wire test (13), and the PAPRICAN/Hobart mixer method (14). The turnaround time of all these methods is too long for process control applications.

The adhesives comprising stickies are polymeric and are, therefore, of high molecular weight (MW). In a secondary fiber facility their concentration should be highest at the pulper and should then fall progressively as they are removed through screening, cleaning, flotation and other operations. The other high-MW chemicals likely to be present at the repulper are residual process polymers. This study is based on the hypothesis that microstickies constitute the majority of the colloidal high-MW material in the front end of the mill. In this paper we show that isolating and measuring this high-MW (operationally defined as >3,000 Da) fraction leads to a microsticking sensor that can be deployed online.

## EXPERIMENTAL

The high-MW material was determined as follows. The sample was filtered and the Total Organic Carbon (TOC) of the filtrate determined with an Ionics Model 1555B Carbon Analyzer (Ionics Inc., Watertown, MA). The filtrate was then ultrafiltered through a membrane (Amicon membranes obtained from Millipore, Inc.) that allowed passage of compounds of MW < 3,000 Da. The TOC of this low-MW permeate was determined. The difference in TOC represents high-MW (>3,000 Da) material.

The tack of the components in a process stream was measured by gently boiling down 700 ml of the filtered sample to 5 ml. A 1-ml aliquot of this concentrate was plated on a metal coupon, which was dried overnight at 25-40°C. The coupon was warmed in water, the water was shaken off, and the tack measured with a ProbeTack PT-500 tack tester (ChemInstruments, Fairfield, OH) at different temperatures as the coupon cooled. Water was used to warm the coupon, since cracks occasionally developed on the surface of the film when it was heated in air. The tack results were interpolated to 40°C to enable comparison across samples.

The model stickie used was BF Goodrich Carbotac 26171 (48.9% total solids).

## RESULTS AND DISCUSSION

The method was validated in the laboratory against a technique proposed by Robert de Jong (15), where macrostickies are screened out and the screenate maintained at 4°C for 2 weeks. The microstickies agglomerate over this period and are screened and weighed. A minor modification was made in that the microstickies were agglomerated by acid-shocking rather than through screening, which eliminated the need for a holding period. Four Carbotac suspensions were acid-shocked with 96% H<sub>2</sub>SO<sub>4</sub> (6 ml/100 ml), the solids settled overnight and filtered. The recovered solids were weighed, and TOC was measured before and after agglomeration. The difference in TOC should correspond to the quantity of solids agglomerated. The results, provided in Table 1, demonstrate an acceptable comparison between the two methods.

The above measurements were repeated in the presence of fiber. Five sheets of Avery return labels (No. 5267) were pulped with 750 g of water in a Waring blender at 50°C for 5 minutes. The slurry was diluted to two gallons and screened through a 100-mesh screen. Three aliquots of the screened sample were subsequently acid-shocked as described above, with TOC being determined before and after acid-shocking. The results, presented in Table 2, translate to an average recovery of 66%. The variability on TOC is small; that for gravimetry is much higher because of the uncertainty involved when small quantities of solids are weighed. Also, fiber debris was collected with the stickies in the gravimetric technique, which would bias the results high. We conclude that the comparison between the two methods is adequate.

In order to demonstrate that the high-MW dissolved components caught on the membrane were, indeed, stickies, samples from various process streams in a newsprint mill were collected and filtered to remove particulates. The filtrate (20 ml) was then ultrafiltered, and water (10 ml) was flushed through the membrane to remove inorganic salts. The membrane surface was then washed with acetone (5 ml) to physically remove the trapped material. The acetone was dried at room temperature to 0.5 ml, transferred to a slide, taken to dryness, and analyzed by reflectance IR using a microscope attachment. No meaningful spectra were obtained when a water sample was processed as a control. The results from the mill samples are provided in Table 3, and show

that stickies constitute a significant fraction of the material caught by the membrane. Of the other components trapped, the inorganics would not appear in our TOC measurement, and the surfactants, defoamers and extractives are likely to be of low molecular weight. Hence, the high MW material isolated is predominantly stickies. Similar results were obtained from a recycle linerboard mill as shown in Table 4.

A final validation of our method was done by comparing the high-MW TOC with the tack of the components in samples collected from various process streams in six mills. We use tack, because it is a direct measure of all adhesive material in the sample. The results, illustrated in the left panel of Figure 1, demonstrate a relationship between tack and high-MW TOC. The curve flattens out at the top probably because the coupon has more than just thin-layer coverage at this point, and the internal bond strength of the residue film contributes to the tack measurement. The intercept is near-zero as would be expected. Only broad generalities can be drawn from a plot of total TOC vs. tack as shown in the right panel of Figure 1. For example, we can say that the samples from mill 1 lie in the right upper quadrant, while those from mill 4 are located in the left lower quadrant of the plot, but further interpretation cannot be supported. The variability in low-MW TOC is evidently high enough to swamp out the contribution of stickies to the total TOC. The relationship with high-MW TOC in Figure 1 is compelling because it is drawn from several sampling points across different types of mills. Clearly, high-MW TOC tracks the tack in the sample. The scatter in Figure 1 (left panel) probably derives from the tack measurements, whose uncertainty approaches 30%. The uncertainty in the high-MW TOC values is less than 10%.

The performance of the microstickies sensor was compared to the more traditional Pulmac shive analyzer using samples taken from various points (repulper to thick stock) along a newsprint mill. The results are illustrated in Figure 2. Note that the Pulmac values drop rapidly after the coarse screen accepts as the macrostickies are removed, whereas the microstickies drop more gradually. The two sets of data are complementary; the Pulmac measures macrostickies, whereas the high-MW method determines microstickies. There is a weak relationship between micro and macrostickies beyond the feed to fine screens as shown in Figure 3. Pulmac data are typically associated with high uncertainty, and the scatter in Figure 3 almost certainly arises from variability in the Pulmac values. Since the population of one size-fraction should be related to that of another, a relationship of the type seen in Figure 3 is expected.

The high-MW method (16) is easily automated. An integrated instrument has been built by Ionics, Inc., and a sample can be automatically processed in eight minutes. We anticipate that the device will have at least four potential applications. One is in process control where a high microstickies count could serve as an early warning for developing problems. We have seen a major difference in repulper microstickies in one mill, which means that we have the ability to flag a bad batch of wastepaper and adjust chemical use and make other process changes to mitigate its effect. A second application lies in testing strategies for evaluating wastepaper batches that enter the mills. A third application is focused on removing microstickies from various process streams. This has been difficult in the absence of an accurate measurement technique. Finally, the sensor can be used as a quality control device. The Pulmac analyzer presently fills this role, and the microstickies sensor would complement the Pulmac instrument. However, the Pulmac device characterizes material that would be screened out in most cases, whereas the mi-

crostickies sensor represents the stickies that are able to traverse the screens and are more likely to enter the system. As such, high-MW TOC may be a more appropriate measure of quality than Pulmac counts.

## ACKNOWLEDGMENTS

This study was funded by the Member Companies of the Institute of Paper Science and Technology.

## REFERENCES

1. Doshi, M.R., Dyer, J.M. (Editors) "Paper Recycling Challenge. Volume 1. Stickies", Doshi & Associates, Appleton, WI (1997).
2. Dyer, J. "Summary of Stickies Quantification Methods", *PaperAge*, 113(9), 28, 30, 32, 36 (1997).
3. Doshi, M.R., Dyer, J.M., Krueger, W.C. "New Stickies Detection Method Can Help You Pick the Right Equipment", *Pulp and Paper*, p. 113 (1984).
4. Darlington, W.B., Blair, W.R. "Quantification of Thermoplastic Stickies with an Image Analyzer", *Progress in Paper Recycling* 4(2), pp. 68-70 (1995).
5. Sisler, G. "Quality Control for Fine Paper Deinking", *Tappi J.* 64(7): 51 (1981).
6. Bittner, M.J. "Stickies Collection and Quantification within a Production Environment", Proceedings of 1994 Pulping Conference, pp. 445-450, TAPPI Press (1994).
7. Blais, S., Chabot, R., Gaudreault, R., Tremblay, C. "Methodology for the Determination of Stickies Concentration in Bleached Kraft Recycled Pulp Using an Interactive Image Analyzer", *Progress in Paper Recycling* 6(2): 85-92 (1997).
8. Hacker, M.P. "Evaluation of Lightweight Contamination in an OCC System", Proceedings of TAPPI Pulping Conference, pp. 455-458 (1991).
9. Venditti, R.A., Andrews, E.K., Chang, H., Heitmann, J.A. "Stickies Related Research at North Carolina State University", in Paper Recycling Challenge, Vol. 1, Stickies, pp. 45-48, Doshi & Associates, Inc. (1997).
10. Elsbey, L.E. "Experiences from Tissue and Board Production Using Stickies Additives", Proceedings of TAPPI Pulping Conference, Book 1, p. 187 (1986).
11. Dykstra, G.M., Hoekstra, P.M., Suzuke, T. "A New Method for Measuring Depositible Pitch and Evaluating Control Agents", Proceedings of TAPPI Papermakers Conference, pp. 327-340 (1988).
12. Ouellette, A.J. "Troubleshooting for Stickies Using the Doshi Method", *Progress in Paper Recycling* 4(2): 85-88 (1995).
13. Cathie, K., Haydock, R., Dias, I. "Understanding the Fundamental Factors Influencing Stickies Formation and Deposition", Proceedings of the 1<sup>st</sup> Research Forum on Recycling, Toronto, ON (published by CPPA), pp. 197-206 (1991).
14. Sithole, B.B., Filion, D., Allen, L.H. "A Laboratory Test to Predict Deposition in Recycled Paper Making", Proceedings of TAPPI Recycling Symposium, pp. 367-371, TAPPI Press (1997).
15. de Jong, R. TAPPI New (CA 960603.01) "Micro-stickies in Process Water: Proposed New Standard Practice", March 3, 2000.
16. Banerjee, S. US patent pending.

<b>Table 1. Recovery of Carbotac by TOC.</b>				
initial solids (g) <sup>1</sup>		agglomerated solids (g)	remaining solids (g)	
added	by TOC		by difference	by TOC <sup>2</sup>
0.753	0.736	0.712	0.041	0.025
0.723	0.719	0.703	0.020	0.032
1.497	1.46	1.473	0.024	0.036
1.489	1.45	1.456	0.033	0.035
<sup>1</sup> in 100 ml for the first two entries and in 200 ml for the next two; <sup>2</sup> TOC was multiplied by 1.8 since carbon constitutes approximately 55% of the acrylate.				

<b>Table 2. Mass balance for the pulped return labels.</b>					
volume (ml)	TOC (ppm) before acid shock	TOC (ppm) after acid shock	$\Delta$ TOC as grams solids <sup>1</sup>	weighed solids recovered (g)	recovery (%)
200.5	185	70	0.0415	0.065	63.8
201.7	185	70	0.0418	0.051	81.9
199.6	185	70	0.0413	0.078	52.9
<sup>1</sup> TOC was multiplied by 1.8 since carbon constitutes 55 % of the acrylate latex.					

<b>Table 3. FTIR-identified contaminants at a newsprint mill.</b>		
alkaline loop	feed to coarse screen	PVAC <sup>1</sup> , extractives, surfactant
	feed to fine screen	PVAC, phthalate, extractive, defoamer
	feed to alkaline flotation cell	PVAC, clay, surfactant, SBR <sup>2</sup> , acrylate
	feed to alkaline DNT washers	PVAC, clay, SBR, defoamer
	alkaline press accepts	clay, PVAC
acid loop	feed to flotation cell	PVAC, defoamer, surfactant
<sup>1</sup> polyvinyl acetate; <sup>2</sup> styrene butadiene rubber		

Table 4. FTIR-identified contaminants at a linerboard mill.	
thickener accepts	extractives, acrylate
fine screens feed	extractives, starch, groundwood
fine screens accepts	retention aid, clarifier polymer, acrylate
fine screens rejects	PVAC <sup>1</sup> , primary clarifier polymer, acrylate, starch, polyvinyl alcohol
reverse cleaner feed	retention aid, clarifier polymer, acrylate
reverse cleaner accepts	groundwood, PVAC
reverse cleaner rejects	PVAC
<sup>1</sup> polyvinyl acetate	

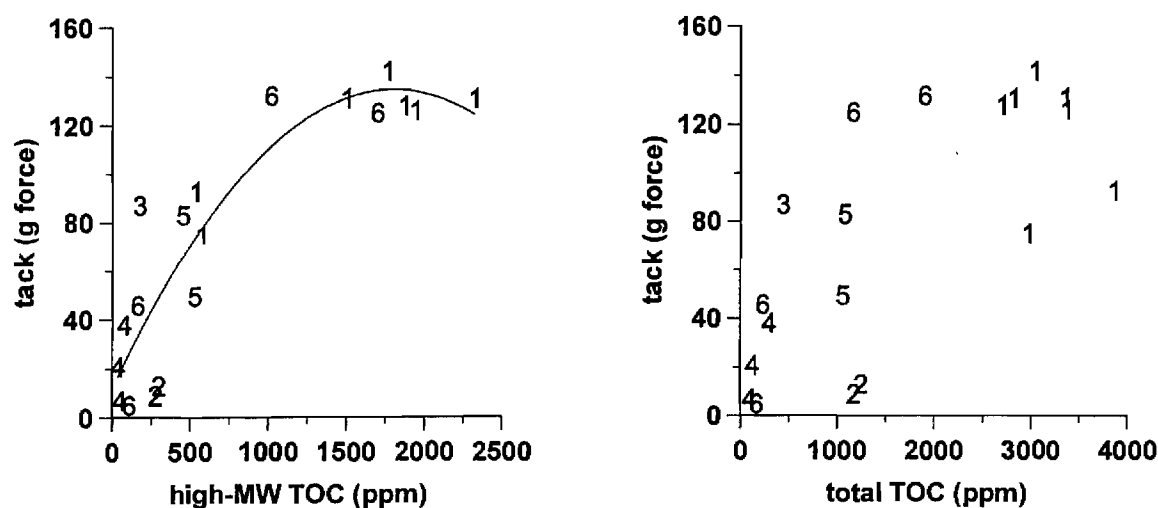
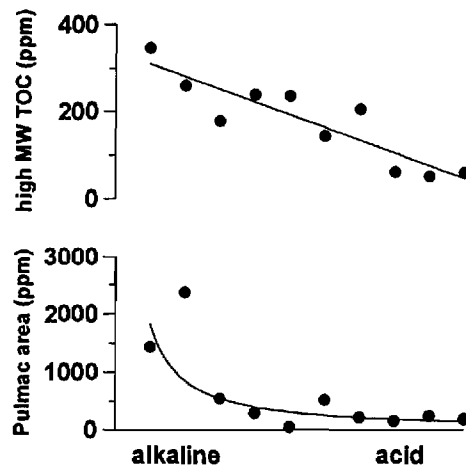
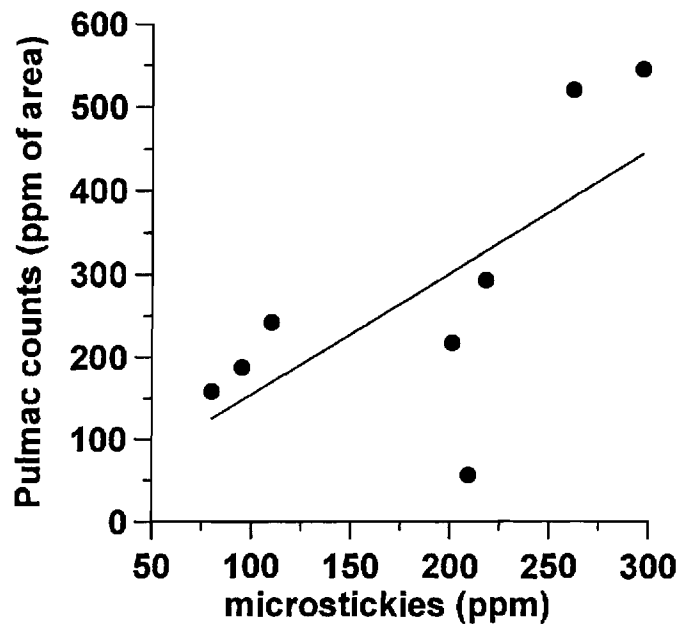


Figure 1: Relationship between tack and high-MW TOC (left) and total TOC (right). Samples were taken from the following types of mills: linerboard/medium (1), linerboard (2), newsprint (3, 5), tissue (4), specialty paper (6).





**Figure 2: Relationship between micro- and macrostickies across a newsprint mill**



**Figure 3: Relationship between micro- and macrostickies at locations beyond the coarse screens.**